EFFECT OF RHENIUM ADDITIONS ON THE
ANISOTROPIC SLIP IN MOLYBDENUM SINGLE CRYSTALS

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THE ANISOTROPIC SLIP IN MOLYBDENUM SINGLE CRYSTALS

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ABSTRACT

The yielding behavior of b.c.c. crystals has been reported to depend asymmetrically on crystal orientation at low temperatures. The asymmetric yielding behavior was assumed by Hirsch and others to be due to the dissociation of the $\frac{a}{2}$ <111> screw dislocations. The present project was initiated to investigate the alloying effect on the asymmetric yielding behavior of b.c.c. crystals. The Mo-Re alloy system was chosen because it was thought that adding Re to Mo would lower the stacking fault energy of Mo and increase the tendency of dissociation of the $\frac{a}{2}$ <111> screw dislocations, hence increase the asymmetry. Single crystals of two Mo-Re alloys, 11 atom percent and 21 atom percent Re, each in three different orientations, were tested under tension at low temperatures. The yield stress and slip band formation of these crystals were found to depend on test temperature, solution concentration, and below room temperature, asymmetrically on crystal orientation. The asymmetric yielding behavior was found to be in reasonably good agreement with the pseudo-Peierls mechanism proposed by Dorn and Mukherjee. However, the degree of asymmetry in

the Mo-Re alloys was found to be less than that for Mo in contrast to the original expectation. It is suggested that Re addition might have increased the stacking fault energy at low Re concentration, then decreased it when Re concentration reached higher value. Moreover, solid-solution softening phenomenon was observed for only two of the three orientations (B and C) of Mo-Re alloys investigated. It seems this is also orientation dependent and might come from the same origin as the asymmetric yielding behavior.

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1. INTRODUCTION

The anisotropic yielding behavior of b.c.c. crystals was observed in beta brass as early as 1928. However, the subject was not investigated intensively until recent years. The existence of the orientattion effect on the deformation behavior at low temperatures has been confirmed in many b.c.c. metals and alloys[2-12]. For a review, see Dorn and Mukherjee[13].

The tensile axis of a b.c.c. crystal is usually represented in the stereographic unit triangle as shown by "o" in Fig. 1. This orientation is defined by the angles λ and X, where λ is the usual Schmid angle between the tensile axis and the slip direction and X is the angle between the plane of maximum resolved shear stress (MRSS) and the $\{\bar{1}01\}$ plane in the same zone of the slip direction rotated clockwise. The angle X is defined between the observed slip plane and the $\{\bar{1}01\}$ plane in the same way as for X. The asymmetric effects can be stated as follows:

1. The yield stress under tension is higher when the crystal is oriented near [011], where X>0 and the $(\bar{2}11)$ $[\bar{1}11]$ mode is the MRSS system, than when it is oriented near [001], where X<0 and the $(1\bar{1}2)$ $[\bar{1}11]$ is the MRSS system [2-10].

2. The slip band formation is also asymmetric[2,3, 6-12]. Under tension it is found to be closer to the MRSS plane for X>0 and deviate toward the $(\bar{1}01)$ plane for X<0 which sometimes totally excludes the operation of the $(\bar{2}11)$ system in the [111] direction.

3. Moreover, the asymmetric behavior is reversed when the sign of the stress is changed from tension to compressions[3, 6-9, 11, 12]. These results suggest the equivalence of the $(\bar{2}11)$ [111] with the $(1\bar{1}2)$ $[1\bar{1}\bar{1}]$ difficult slip, which are antitwinning systems, and also the equivalence of the $(1\bar{1}2)$ [111] with the $(\bar{2}11)$ $[\bar{1}\bar{1}\bar{1}]$ easy slip which are twinning systems. This indicates that the slip is difficult when the atoms in the core of the dislocations move in the antitwinning direction and easy when the atoms move in the twinning direction.

It has been thought for some time that the asymmetric yielding of b.c.c. crystals was due to the dissociation of screw dislocations[14-16]. However, it is also known that experimentally no dissociation or stacking fault has been observed. Theoretical calculations[17-18] have also shown that no metastable large stacking fault exists in b.c.c. metals and dissociations are limited to at most a few Burgers vectors. Recently, Dorn and Mukherjee[13] suggested that although no true dissociations of the screw dislocations take place, a tendency toward splitting exists which leads to a decrease in the core energy of the dislocation with a corresponding asymmetric disposition of the core atoms. Therefore, the Peierls stress for the $(\bar{2}11)$ [111] antitwinning system be expected to exceed that for the $(\bar{1}12)$ $[\bar{1}11]$ twinning system. The asymmetric arrangement of the core atoms was assumed to be modified by the applied stress. For $X>0$ the modification would move the atoms toward an arrangement which is more difficult to slip whereas the opposite takes place for $X<0$. With this concept, they were able to modify the Dorn-Rajnak model of the Peierls mechanism so as to account for the asymmetric slip behavior of

b.c.c. crystals at low temperatures. A systematic investigation on Mo[10] gave results in reasonable agreement with their predictions.

It was hoped in this investigation to change the stacking fault energy of a pure metal by alloying so as to change the dissociation tendencies of the screw dislocations and to observe the change of deformation behavior. The Mo-Re system was chosen because previous work on this alloy[20,21] has shown that at higher concentrations in the solid solution range (above about 25 atom per cent Re) twinning is the predominant mode of deformation at low temperatures. It was hence thought that Re additions might lower the stacking fault energy of Mo. Futhermore, deformation twins [22,23] and stacking fault groups[24] have been observed in Mo-Re alloys using transmission electron microscopy.

2. EXPERIMENTAL TECHNIQUES

Single crystals of Mo-Re alloys, denoted Mo-11 Re and Mo-21 Re, were grown by melting together commercially pure Mo and Re rods in an electron beam zone refining aparatus. In order to maintain a homogeneous distribution of Re the direction of zoning was reversed after each pass. Chemical analyses showed that Mo-11 Re contained 11.3 atom per cent Re and less than 200ppm interstitial impurities, mainly C, O and N. Mo-21 Re contained 20.9 atom per cent Re and less than 300 ppm interstitial impurities. The distribution of Re in the crystal was checked by electron microprobe analysis and found to be uniform both longitudinally and crosswise to within the limit of the method (13%).

Three orientations, A, B and C, were selected as shown in Fig. 2a. These have $\chi = 45^{\circ}$ and $\chi = -30, + 28^{\circ}$ and 0° respectively. Tensile specimens, each having a 0.7 inch gage length and 0.085 inch square

cross section were machined by grinding. The gauge surfaces of the
specimens were selected such that one set of the surfaces, called
side faces, were parallel to the maximum stressed <111> direction as
shown in Fig. 2b. The specimens were electropolished in a solution
containing equal parts of methyl alcohol (CH_3OH) and sulfuric acid
(H_2SO_4) at $\sim 0°C$ and then annealed in He atmosphere at $2000°C$ for
24 hours.

Tensile tests were performed at various constant temperatures
from about $20°K$ to $900°K$ in an Instron testing machine at a strain
rate of $\sim 5 \times 10^{-5}$/sec. The yield stress was determined at an offset
of 0.001 strain from the modulus line. Specimens were removed at
strain of 0.002 and 0.05 for slip trace observations using the
Normaski interference contrast technique on a Ziess Ultraphot Micro-
scope. Operative slip systems were identified by the two-surface
slip trace analysis method.

3. EXPERIMENTAL RESULTS

1. Tensile Yield Stresses

Deformation of the Mo-Re alloy crystals in this investigation
was found to take place exclusively by slip processes. No evidence
of twinning was observed. For Mo-11 Re crystals, plastic strain
was measured even at the lowest temperature tested, i.e. about
$20°K$. The Mo-21 Re alloy, however, became very brittle at
temperatures below liquid nitrogen temperature, and no meaningful
yield stress could be recorded.

The yield stress at low temperature is usually considered to
consist of two parts, a thermal component, called effective stress
(σ^*), and an athermal component, which is believed to be due to
the long range internal stress of the crystal and varies with
temperature only as does the shear modulus of elasticity.

The athermal stresses, taken at the temperature independent level of the yield stresses at higher temperatures, were measured to be about 13×10^8 dynes/cm^2 for the Mo-11 Re alloy and about 16.5×10^8 dynes/cm^2 for the Mo-21 Re alloy. Combining the athermal stress of about 1 to 2×10^8 dynes/cm^2 reported for mo[10], it seems that the increase in athermal stress varies approximately with the square root of the Re concentration. The effective stress, which is obtained by subtracting the modulus-corrected athermal stress at the test temperature from the experimentally determined tensile yield stress, is then plotted versus temperature in Fig. 3 for both Mo-11 and Mo-21 Re alloys.

The results clearly indicate that the effective stress of Mo-Re alloys is dependent not only on temperature but also on the crystal orientation and alloy composition. The difference in the temperature dependence of the effective stress with respect to crystal orientation is particularly insteresting for orientations A and B, since both have {112} as the MRSS planes. For orientation A, $X = \sim 30^\circ$, the maximum stressed $(\bar{2}11)$ [111] system is the antitwinning sense. Consequently the deformation behavior is seen to be asymmetric and slip in the antitwinning sense requires the higher stress. The asymmetric deformation behavior will be further demonstrated when the slip geometry is discussed in the next section.

The dependence of effective stress on alloy composition is shown in Table I. The effective stresses at 77°K for Mo-11 Re and Mo-21 Re are listed together with those reported on Mo[10] in Table I. Also listed are the ratios of the effective stress which are used as an indication of the asymmetric effect. The asymmetric effect is highest for Mo and lowest for Mo-11 Re alloy. In contrast to the original expectation, the asymmetric effect at first decreases

with Re addition, but then increases as the Re concentration is raised further. The effective stress increases monotonically with Re concentration for orientation A. Yet for orientations B and C, while Mo-21 Re alloy has the highest effective stresses, Mo-11 Re alloy has the lowest. Similar alloy softening phenomena have been reported on Ta alloys,[24, 25] Mo-Re alloy[21] and recently on Fe alloys,[26, 27] etc. The interesting point of the present results is that alloy softening depends strongly on crystal orientation.

2. Slip Geometry

The general appearance of slip bands on both front and side faces is shown in Fig. 4. The slip bands are straight and sharp on the side face but wavy and rather ill defined on the front face. The slip process seems to be relatively homogeneous through the gauge section of the specimens. At the beginning of deformation, e.g. $\varepsilon \sim 0.0002$ more than one slip system was detected for orientations A and C, similar to the result reported for Mo[10]. As the crystals deformed further, (e.g. $\varepsilon \sim 0.005$) one slip system seems to be predominant.

The operative slip Burgers vector, as determined by the two face slip trace analyses and the rotation of the tensile axis was found to be the highest stressed $\frac{a}{2}$ <111> Burgers vector, i.e. for orientation A, $\frac{a}{2}$ [$\bar{1}$11], and for orientations B and C $\frac{a}{2}$ [111], regardless of composition. The exception was orientation C of Mo-21 Re alloy at 77°, for which slip took place via $\frac{a}{2}$ [$\bar{1}$11] in stead of $\frac{a}{2}$ [111].

The choice of the operative slip plane was complicated by its dependence on crystal orientation, composition and even the test temperature. Micrographs of the slip band on a front face of the

Mo-11 Re alloy are shown in Figs. 5A, 5B, and 5C for orientations
A, B and C respectively. The results are summarized as X vs. T
curves in Fig. 6. For orientations A and C the operative slip planes
were (1$\bar{1}$2) and $\bar{1}$01) respectively over the whole temperature ranges
investigated. Those are the MRSS planes in these samples. However,
in orientation B slip took place on the ($\bar{1}$01) plane at test tempera-
tures below 156°K rather than on the maximum stressed ($\bar{2}$11) plane.
This latter slip plane was not found until above about 375°K. There
was a transition temperature range between 156°K and 375°K as the
operative slip lane changed from ($\bar{1}$01) toward ($\bar{2}$11).

For Mo-21 Re alloy crystals slip band micrographs of orientations
A, B and C are shown in Figs. 7A, 7B and 7C respectively. The opera-
tive slip planes are plotted as X vs. T curves in Fig. 8. It is seen
that slip took place on the (1$\bar{1}$2) plane for orientation A at all
temperatures investigated and on the ($\bar{2}$11) plane for orientation B
with some deviation toward the ($\bar{1}$01) plane between 117°K and 195°K.
As for orientation C, slip took place on the ($\bar{1}$01) plane and in the
[111] direction above 296°K and then deviated toward the ($\bar{1}\bar{1}$2)
plane in the same direction as the test temperature was lowered.
But at 77°K, the slip system was found to be in the (1$\bar{1}$2) [$\bar{1}$11] mode.

The principal slip systems at 77°K for Mo-Re alloys and Mo are
summarized in Table 4. The asymmetric slip behavior is obvious. For
Mo and Mo-11 Re alloy crystals, the ($\bar{2}$11) [111] mode was not observed
at this temperature even if orientation B, which has the highest
effective stress, was especially chosen to have the ($\bar{2}$11) [111]
mode as the maximum stressed system. Therefore, the critical
resolved shear stress (CRSS) to operate the ($\bar{2}$11) [111] system, which
is an antitwinning system, must be very high for Mo and Mo-11 Re
alloy, while the (1$\bar{1}$2) [111] system, which is a twinning system, is

operative at much lower effective stresses. However, for Mo-21 Re alloy the ($\bar{1}$01) [111] mode was not observed at 77°K. Instead of the maximum stressed ($\bar{1}$01 [111] mode for orientation C, the (1$\bar{1}$2) [$\bar{1}$11] mode was found to be operative. Consequently, the results indicate that the (1$\bar{1}$2) [$\bar{1}$11] mode, which is a twinning system, is easier to operate for this alloy. The effective stress for orientation B is also higher than those of orientations A and C. The striking result here is that the CRSS calculated from the effective stress of orientation C is lower than that from orientation A, even when orientation C has higher X angle (-10° versus -30°). This will be further discussed in the following section.

IV. Discussion

It is clear from the experimental results that the deformation behavior of Mo-Re alloys is asymmetric, at least at the lower test temperatures, i.e., below about room temperature. Qualitatively, the asymmetric behavior of Mo-Re alloys is similar to that of Mo and in agreement with the Dorn and Mukherjee's model[13]. However, quantitively, it seems that the asymmetric effect decreased from Mo to Mo-11 Re alloy and then increased from Mo-11 Re alloy to Mo-21 Re alloy. As stated in the introduction, the asymmetric behavior of b.c.c. crystals is explained by Dorn and Mukherjee[13] as due to the asymmetric core structure of the screw dislocation. They suggest that the apparent Peierls stress may be higher for the dislocation to move in the antitwinning direction than in the twinning direction on the {112} plane. Furthermore, the asymmetrical arrangement of the atoms in the core of the dislocation is assumed to be modified by the applied stress. Asymmetric factors, $A_{(hkl)}$, are then defined to account for these modifications in the following equations.

$$\tau_{\rho(\bar{1}01)} = P_{(\bar{1}01)} + A_{(\bar{1}01)}\sigma^* \cos\lambda \sin\lambda \sin 3\chi \ldots\ldots\ldots(1)$$

$$\tau_{\rho(1\bar{1}2)} = P_{(1\bar{1}2)} + A_{(1\bar{1}2)}\sigma^* \cos\lambda \sin\lambda \sin 3\chi \ldots\ldots\ldots(2)$$

Where $\tau_{\rho(hkl)}$ is the Peierls stress on the (hkl) plane, $P_{(hkl)}$ represents the Peierls stress when either χ or σ^* is equal to zero, and λ and χ should be chosen in accord with the operative Burgers vector. For Mo-11 Re alloy, $A_{(\bar{1}01)}$ was calculated to be 0.06 which is relatively samll compared with $A_{(\bar{1}01)} = 0.263$ reported on Mo[10]. $A_{(1\bar{1}2)}$ was found to be -0.25 for Mo-21 Re alloy as compared with $A_{(1\bar{1}2)} = 0.125$ for Mo[10]. The negative sign of the $A_{(1\bar{1}2)}$ for Mo-21 Re alloy comes from the fact that the CRSS on the $(1\bar{1}2)$ plane was smaller when calculated from the effective stress of orientation C, $\chi = -10°$ than from orientation A, $\chi = -30°$. This result seems unrealistic from the model's suggestion. However, the ratio of the effective stresses for orientations B and C (Table 1) is indeed bigger which shows an increase in the asymmetry. $A_{(101)}$ for Mo-21 Re alloy and $A_{(112)}$ for Mo-11 Re alloy are not calculated due to insufficient data.

The present investigation was initiated on the assumption that Re addition might lower the stacking fault energy of Mo for it was shown to have promoted twinning mode of deformation[20, 21]. If one thinks the asymmetric deformation behavior is originated from the asymmetric dissociated core structure, then a decrease in the stacking fault energy should lead to an increase in the asymmetric behavior, since the degree of dissociation of the dislocation is very much dependent on the stacking fault energy. However, ex-perimental results from this investigation have indicated that the asymmetric behavior was at first decreased and then increased again due to Re additions. This phenomenon might lead one to suggest

that the stacking fault energy was increased at low Re concentration and then decreased when the Re concentration becomes higher. In fact, deformation by twinning was not observed during the course of this investigation. Those previous works on Mo-Re alloy crystals[20, 21] have also indicated that twinning mode was not observed until Re concentration reached about 25 atom per cent or more. A similar phenomenon was reported on Nb alloys by Koss[28]. He found that alloying 6 atom. percent W in Nb crystal increased the asymmetry of yielding, yet his Nb crystals, which contained about 450 ppm interstitial impurities, showed negligable asymmetry compared to the asymmetric deformation reported on ultra-high purity Nb[6,7], containing only about a few ppm interstitial impurities. Although his Nb crystal contained interstitial impurities instead of substitutional solute atoms, the effects seem to be analogous.

For b.c.c. alloys, the solid solution softening at low temperatures is a fairly general phenomenon[21, 24-27]. This softening can occur in either substitutional or interstitial[29] alloys. Although it has been suggested that association between substitutional and interstitial solutes or between interstitials of the same or different types, called scavenging effect, can cause solid-solution softening, recent investigations seem to prefer a modification of the Peierls mechanism through either a decrease of the Peierls energy[21, 24, 25] or a reduction of the kink nucleation energy[26, 27].

The present results showed that the Peierls stresses are lower for orientations B and C of Mo-11 Re alloy than Mo indicating a lowering of the Peierls energy due to Re addition. This is in agreement with the results of Davisdon and Brotzen[21]. Solid solution softening was not observed for orientation A indicating that this phenomenon might be also orientation dependent. Once again, if one

thinks that the high Peierls energy as well as the asymmetric

deformation behavior of b.c.c. crystals are due to the asymmetri-

cally dissociated core structure of the dislocations as suggested

by Dorn and Mukherjee[13] then a lowering of the Peierls stress

could mean an increase in the stacking fault energy. This is in

line with the foregoing discussions where the reduction of the

asymmetric deformation behavior was thought to suggest an increase

in the stacking fault energy. Present knowledge on the effect of

solute atoms on the stacking fault energy as well as the detailed

core structure of dislocations does not seem to be able to provide

any quantitative analysis.

SUMMARY

1. The yielding behavior of single crystals of two Mo-Re alloys,

   11 atom percent and 21 atom percent Re, each with three different

   orientations were investigated under tension and the results

   were compared with those of Mo reported by Lau et al.[10].

   The deformation of these crystals was found to depend on tempera-

   ture, alloy composition and below room temperature, asymmetri-

   cally on crystal orientation.

2. At low temperatures, where asymmetry was observed, slip occurred

   on both $(1\bar{1}2)$ $[\bar{1}11]$ and $(\bar{1}01)$ $[111]$ systems, but never on the

   $(\bar{2}11)$ $[111]$ mode for Mo-11 Re alloy crystals, even with the

   most favorable orientation, which is similar to the results

   of Mo. However, for Mo-21 Re alloy crystals slip did take place

   by the $(\bar{2}11)$ $[111]$ mode, but never occurred by the $(\bar{1}01)$ $[111]$

   mode, instead, the $(1\bar{1}2)$ $[\bar{1}11[$ mode was more favored. The

   Yield stress for slip by the $(\bar{2}11)$ $[111]$ antitwinning mode was

   found to be much higher than that for the $(1\bar{1}2)$ $[\bar{1}11]$ twinning

mode. This asymmetric behavior is in agreement with the pseudo-Peierls mechanism suggested by Dorn and Mukherjee[13].

3. Although the asymmetric slip behavior is clear and evident for Mo-Re alloys, the degree is less than that for Mo. This is in contrast to the original expectation as Re additions were thought to lower the stacking fault energy of Mo and to increase the asymmetric effect. Thus, it is suggested from present results that Re addition migh have increased the stacking fault energy at low Re concentration, then decreased it when Re concentration became higher.

4. Solid-solution softening phenomenon was observed for orientations B and C of Mo-Re alloys but not for orientation A. Thus the solid solution softening is also orientation dependent and might be related to the asymmetric slip behavior.

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